भारतीय मानक
Indian Standard

IS 7686 : 2020

3 (एन, एन-डाईइथाइल) एमिनोफिनॉल की विशिष्टि

(पहला पुनरीक्षण)

Specification for 3 (N, N-Diethyl) Aminophenol

(First Revision)

ICS 71.080.90

भारतीय मानक ब्यूरो
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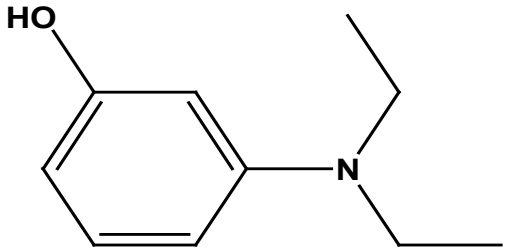
January 2020 **Price Group 4**

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

3 (N, N-Dicthyl) amino phenol ($C_{10}H_{15}ON$) is an intermediate used in the manufacture of dyestuffs. It has the following structural formula:



3-(N,N-DIETHYL) AMINOPHENOL

(Molecular Mass 165.2)

(CAS No 91-68-9)

This standard was first published in 1975. Considering to the fact that since 1975 specification of 3-(N:N-diethyl) amino phenol has not been updated, the Committee has decided to update this specification to incorporate latest instrumentation methods for more precise determination of purity and impurity profile by HPLC.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR 3 (N, N-DIETHYL) AMINOPHENOL

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 3 (N, N-diethyl) aminophenol.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
5299 : 2001	Methods of sampling and tests for dye intermediates(*first revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
1070 : 1992	Reagent grade water — Specification *(third revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white to light brown, moist crystalline solid.

3.2 The material shall be soluble in alcohol, ether, sodium hydroxide and hydrochloric acid.

3.3 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Gross, net and tare mass.

4.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Number of Tests

Tests for the determination of crystallizing point and assay shall be conducted on each of the individual samples.

5.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of this standard, if the test results as obtained in **5.2** satisfy the corresponding requirements given in Table 1.

Table 1 Requirements for 3 (N, N-Diethyl) Aminophenol

(*Clauses* 3.3, 5.3 and 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C (on dry basis), *Min*	69.5	**A-2**
ii)	Purity by CV, percent, *Min*	98.0	**A-3**
iii)	Purity by HPLC, *Min*	98.0	**B**
iv)	Salt content as NaCl, percent, *Max*	0.5	**C**
	NOTE — 3 (N,N-diethyl) aminophenol has a storage life of about three months from the date of manufacture. On prolonged storage, the material becomes sticky and the crystallizing point is considerably reduced.		

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed in Table1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1 and *Clause* 3.3)

METHODS OF TEST FOR 3 (N, N-DIETHYL) AMEMOPHENOL

A-1 PREPARED SAMPLE

Dry the material in a vacuum oven at 45°C to constant mass and transfer immediately into a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

A-2 DETERMINATION OF CRYSTALLIZING POINT

Determine the crystallizing point of the *prepared sample* (*see* **A-1.1**) as prescribed in **8** of IS 5299.

A-3 ASSAY

A-3.0 Outline of the Method

The purity of the material is determined by coupling it with p-nitrobenzene diazonium chloride.

A-3.1 Reagents

A-3.1.1 *Dilute Hydrochloric Acid,* approximately 5 N.

A-3.1.2 *p-Nitrobenzene Diazonium Chloride Solution* — 0.1 N. Dissolve 34.5 g of p-nitroaniline in 100 ml of concentrated hydrochloric acid and 100 ml of water by heating and dilute to one litre with warm water. Add 200 ml of 0.25 N p-nitroaniline solution to a 500 ml volumetric flask cooled to 5°C. Add 50 ml of 1 N sodium nitrite solution rapidly which has been cooled to 5°C. Dilute the resultant solution to 500 ml with water which has been cooled to 5°C. It should give a positive test for nitrous acid when tested with a starch-iodide paper and is ready for use after standing for one or two minutes. Store the solution in an ice-bath in the dark. The solution should be practically colourless and not yellow and it should not be more than slightly turbid. Do not use the solution after standing for more than 5 h. Standardize the solution freshly before use.

A-3.1.3 *Tetrazodianisiditte Solution* — Dissolve 2 g of dianisidine hydro-chloride in 7 ml of hydrochloric acid. Heat if necessary. Cool to 0°C and titrate with 1 N sodium nitrite solution to just completion of reaction. Make up the solution to 100 ml in a volumetric flask. Store this solution in an amber-coloured bottle in a cool place.

A-3.1.4 *H-Acid* (1-*Amino-8~Hydroxy-Naphthalene-3,6~Disulphonic Acid) Indicator Solution* — Dissolve 0.5 g of H-acid in 100 ml of 1 percent sodium carbonate solution.

A-3.2 Procedure

Weigh accurately about 5 g of the prepared sample (*see* **A-1.1**) into a 500 ml beaker. Add about 200 ml of water and just enough of hydrochloric acid to dissolve the sample. Transfer this solution to a 500 ml volumetric flask quantitatively and dilute up to the mark with water. Take 50 ml of this solution into a one 1itre beaker. Add about 45 g of sodium acetate (hydrated) crystals and cool the mixture externally to 10°C. Titrate against p-nitrobenzene diazonium chloride solution taken in a jacketted-burette through which ice-cold water is circulated. As the titration progresses, place a drop of the reaction mixture on Whatman No. 1 filter paper (or equivalent) and touch the runout with a drop of tetra-azodianisidine solution. If there is any amount of uncoupled 3 (N, N-diethyl) aminophenol in the reaction mixture, there will be a colour development at the junction of the two solutions. This colour fades progressively and at a stage it disappears completely. Now touch the runout with the H-acid solution. A pink colour which persists for 5 mins marks the end point.

A-3.3 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 165}{M}$$

Where,

V = Volume in ml, of p-nitrobenzene diazonium chloride solution required for the sample;

N = Normality of the diazonium chloride solution; and

M = Mass in g, of the material taken for the test.

ANNEX B

(*Table* 1 and *Clause* 3.3)

TO DETERMINE PURITY OF 3(N,N-DIETHYL)AMINOPHENOL BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-1 OBJECTIVE

To determine purity of 3 (N,N-diethyl) aminophenol by high performance liquid chromatography.

B-2 APPARATUS

Binary gradient liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-3 COLUMN

C18 100A 250 × 4.6mm, 5µm

B-4 REAGENT

a) Acetonitrile, HPLC grade;
b) Water, HPLC grade;
c) Di-sodium hydrogen orthophosphate;
d) Ammonium di hydrogen orthophosphate; and
e) 3(N,N-diethyl)aminophenol

B-5 STANDARD PREPARATION

Weigh accurately 0.0500 gm 3 (N,N-diethyl) aminophenol in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

B-6 SAMPLE PREPARATION

Weigh accurately 0.0500 gm sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

B-7 BUFFER PREPARATION

Take 0.5750 gm ammonium di-hydrogen ortho-phosphate and 0.7 000 gm di-sodium hydrogen orthophosphate in 1 litre volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water.

B-8 FLOW RATE: 1.00 ml/min

B-9 MOBILE PHASE:

Actonitrile	:	Buffer
45	:	55

B-10 COLUMN OVEN TEMPERATURE: 40°C

B-11 INJECTION VOLUME: 10µl

B-12 RUN TIME: 15 min

B-13 WAVE LENGTH: 254 nm

B-14 PEAK TIME: 3(N, N-diethyl) aminophenol - 7.25 min

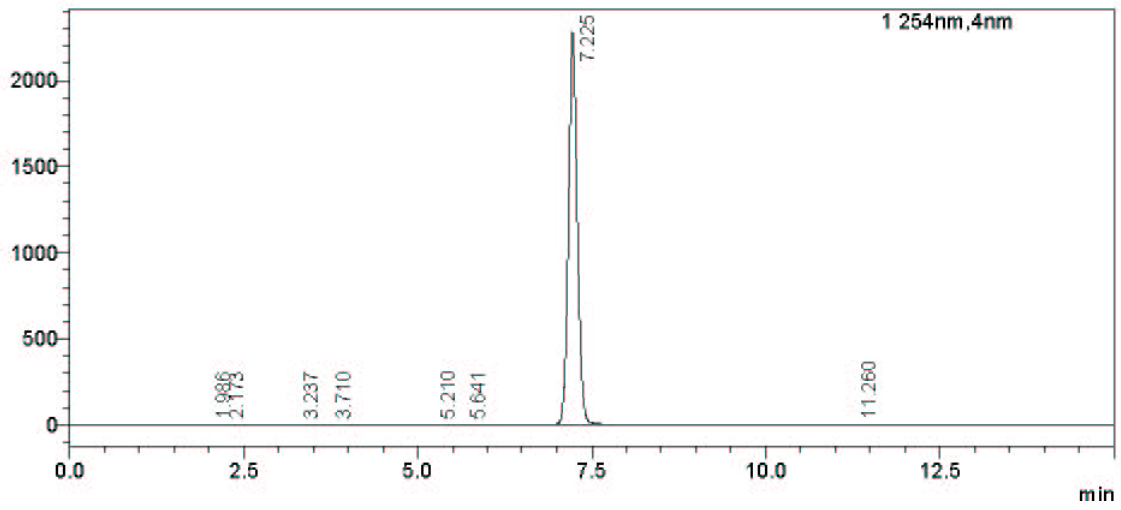


FIG. 1

B-15 CALCULATION

Calculate the peak area of individual constituent pertaining to 3(N,N-diethyl) aminophenol on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure 3(N,N-diethyl) aminophenol.

Percent of 3(N,N-diethyl) aminophenol

$$= \frac{A2 \times V1 \times W1 \times B2}{A1 \times V2 \times W2 \times B1} \times 100$$

Where,

A1 = Area of STD 3 (N,N-diethyl) aminophenol;

V1 = Injection volume of STD 3 (N, N-diethyl) aminophenol;

W1 = Weight of STD 3 (N,N-diethyl) aminophenol;

B1 = Total volume of STD 3 (N, N-diethyl) aminophenol;

A2 = Area of 3 (N,N-diethyl) aminophenol peak in sample;

V2 = Injection volume of sample;

W2 = Weight of sample; and

B2 = Total volume of sample.

ANNEX C

(*Table* 1 and *Clause* 3.3)

C-1 TO DETERMINE SALT CONTENT AS SODIUM CHLORIDE (NACL) BY TITRATION

C-1.1 Requirement

a) Paper boat (butter paper/glossy paper);

b) Wash bottle;

c) 100 ml volumetric flask;

d) 250 ml conical flask;

e) 50 ml volumetric pipette;

f) 50 ml burette;

g) Magnet and magnetic stirrer;

h) 0.1 N silver nitrate solution [standardized] [$AgNO_3$]; and

j) 0.1 Percent potassium chromate indicator [K_2CrO_4].

C-1.2 Testing Procedure

C-1.2.1 *Points*

a) Take accurately approx weight 1.0 gm of sample in paper boat. Transfer it in 100 ml volumetric flask.

b) Add approx 50 ml distilled water. Shake well and finally make 100 ml.

c) Take 50 ml with volumetric pipette from above solution.

d) Add 5 to 6 drop of 0.1percent potassium chromate indicator.

e) Fill the burette with the 0.1 N $AgNO_3$ up to the mark.

f) Add 0.1 N $AgNO_3$ solution to it from the burette gradually, till the solution become yellow to brownish.

g) Note the burette reading.

C-1.2.2 *End Point,* Yellow to brownish.

C-1.3 Calculation

Percent of salt content as NaCl (w/w)

$$= \frac{\text{B.R.} \times \text{Normality of } AgNO_3 \times 58.5 \times 100}{\text{Weight of sample (In gm)} \times 10 \times 50}$$

Where,

B.R. = Burette reading.

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
ICT, Mumbai	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited - Atul – Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K. Astik(*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Smt Maitri Vyas (*Alternate*)
Ministry of Chemicals & Fertilizers – Department of Chemicals & Petrochemicals, New Delhi	Shri Jasbir Singh
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National Test House(er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr Jaychandran M. Nair

Organization	*Representative(s)*
The Bombay Textile Research Association, Mumbai	Dr Ashok N. Desai
The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar
BIS Director General	Shri V. K. Diundi, Scientist 'F' and Head (PCD) [Representing Director General BIS (*Ex-officio*)]

Member Secretary

Shri Chandrakesh Singh

Scientist 'C' (PCD), BIS

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BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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